ULTRA-HIGH RESOLUTION SMALL-ANGLE NEUTRON SCATTERING (USANS)

A perfect crystal diffractometer (PCD), shown in Fig.1, for ultra-high resolution small-angle neutron scattering (USANS) measurements is now in operation at the thermal neutron beam port, BT-5. The PCD increases the maximum size of features accessible with the NCNR's 30 m long, pinhole collimation SANS instruments by nearly two orders of magnitude, from $\approx 10^2$ nm to 10^4 nm.

The PCD is a Bonse-Hart type instrument with large triple-bounce, channel-cut Si (220) crystals as monochromator and analyzer. The perfect crystals provide high angular resolution while the multiple reflections suppress the "wings" of the beam profile, improving the signal-to-noise ratio to values comparable to that obtained with pinhole instruments. This technique, widely utilized for x-rays for many years, has only recently been successfully adapted for neutrons [1] as dynamical diffraction effects arising from the deep penetration of neutrons in thick perfect crystals have become better understood. Neutrons can, in effect, propagate through a thick crystal, and then reflect from the back-face of the crystal. The geometry of this second diffraction path allows part of the beam to bypass the second and third reflections. The design of the NCNR's PCD [2] successfully eliminates the single reflection path by adding shielding along the middle of the long face of each crystal between the first and third reflections (see inset in Fig. 2). The additional shielding reduces the wings in the rocking curve by two orders of magnitude, resulting in a signal-to-noise ratio of 10^5 at a minimum scattering vector $Q = 0.0005$ nm^{-1}. Figure 2 shows typical rocking curves with and without shielding of the deleterious back-face reflection. The beam flux obtained for smaller samples is 3000 cm^{-2} s^{-1}, while the maximum intensity obtained is 15 000 s^{-1} when using the maximum 3×5 cm^2 beam size. The mainly fast neutron flat background (≈ 0.15 s^{-1}) found at large angles is independent of beam size. The beam intensity will increase somewhat when the present perfect crystals are replaced by ones with a wider channel, and a gap in the middle of the long face, in order to increase the beam width to 4 cm with no contamination from single back-face reflections.

The measurement range of the PCD overlaps that of the NCNR's 30 m SANS instruments. Together they probe structure in materials over four orders of magnitude, from $\approx$1 nm to 10^4 nm. Combined measurements on these instruments will enable fuller characterization of hierarchical and highly anisotropic microstructures in materials, for example in fiber or clay impregnated nanocomposites. The PCD is part of the NIST/NSF Center for High Resolution Neutron Scattering (CHRNS) with up to two-thirds of the available beam time to be allocated by the NCNR's Program Advisory Committee to scientists and engineers who submit proposals for peer review.

The PCD USANS instrument can accept any ancilliary sam-

Photography by L. A. Shuman

FIGURE 1. NCNR's Derek Ho (top) and John Barker load a sample at the PCD. The triple-bounce analyzer is visible in the center foreground of the picture.

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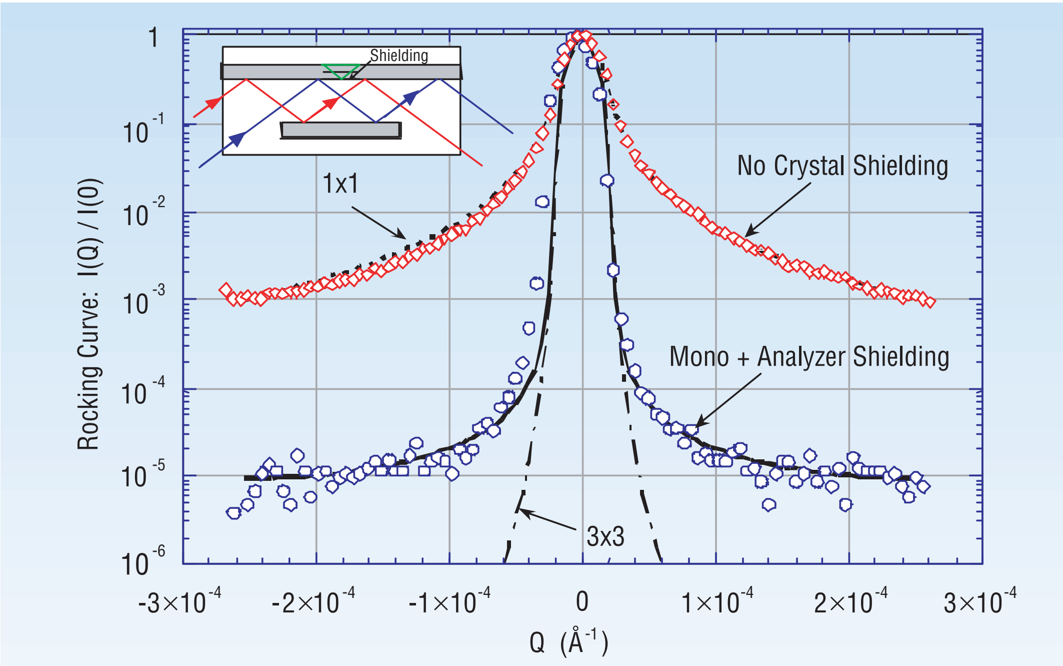

FIGURE 2. Rocking curves measured for the PCD USANS instrument. The diamond symbols are data taken before adding shielding to block back-face reflections from the triple-bounce, channel-cut monochromator and analyzer crystals. The circle symbols are data taken after adding such shielding. The dash-dot curve is the theoretical profile for a pair of triple-bounce perfect crystals. The solid line is the weighted sum of the theoretical profiles for 3x3 and 1x1 rocking curves, with weighting factors of 0.998 and 0.002, respectively. The inset shows a schematic diagram of a channel-cut crystal with the shielding needed to remove the single reflection path from the back-face of the crystal.

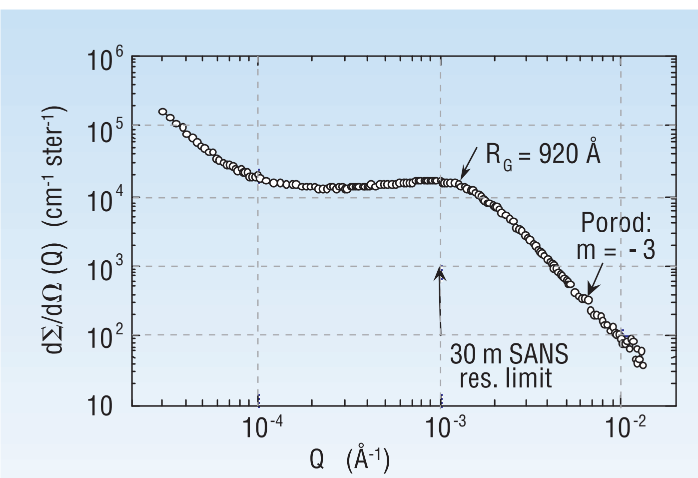

FIGURE 3. First USANS measurement obtained using new PCD instrument. The sample is a 2.5 cm thick commercially obtained poly(tetrafluoroethylene) plate. Scattering is predominately from residual sub-micron size pores (≈ 0.2 % by volume) that survive the extrusion processing of the material.

ple environment equipment that is used on the 30 m SANS instruments. Larger liquid sample cells and a dedicated two-position heating block (30 °C to 400 °C) are currently being designed to utilize the larger available beam size.

The first USANS measurement was made in May 2000 on a commercially obtained poly(tetrafluoroethylene) plate. The slit-smeared data are shown in Fig. 3. The data easily overlap the accessible *Q*-range the 30 m SANS instruments. Examples of material systems studied so far are: pigment aggregation in paint, clay aggregation structures in various solutions and polymer melts, pores in copper, hydrides in uranium, and large scale structures in controlled pore glasses.

REFERENCES

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[2] A.R. Drews, J.G. Barker, C.J. Glinka, M. Agamalian, Physica B **241-243**, 189 (1998).